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Richard Zimmermann

APPLICATION FOR  
UNITED STATES LETTERS PATENT

SPECIFICATION

TO ALL WHOM IT MAY CONCERN:

Be it known that I, Klaus Klenk a citizen of Germany, residing at  
Norder Allee 7, 24939 Flensburg, Germany, have invented a new and useful  
A PROCESS FOR THE RECYCLING OF PET CONSTITUENTS AND AN  
APPARATUS FOR CARRYING OUT THE PROCESS, of which the following is  
a specification.

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**A process for the recycling of PET constituents and an apparatus for carrying out the process**

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**Description**

This invention relates to a process according to the precharacterising clause of claim 1 and to an apparatus according to the precharacterising clause of claim 13.

In PET washing and treatment installations which are known in practice, PET flakes (ground pieces of plastics, of size about 5 to 10 mm) are treated in washing centrifuges or in stage treatment cleaning devices at high speeds of rotation and under considerable friction, with the addition of relatively small amounts of water. Due to the very high power which is used for a relatively short treatment time, the flakes are subjected to severe mechanical damage, and small pieces together with cracked and split flakes are produced, which can cause problems in further stages of the treatment process. These small pieces have to be removed, which is costly and which reduces the yield. Other disadvantages are that the surface of the flakes is only cleaned unsatisfactorily, namely to a shallow depth, bonding adhesives cannot reliably be removed, and extraneous substances which have diffused into the surface cannot satisfactorily be dissolved out or dissolved away. These substances can only satisfactorily be removed in further stages of the treatment process, in a different manner which involves a high level of additional cost.

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In a process which is known from EP 0 304 667 A, fine material and paper is separated from the comminuted material in an air classifier, before dust and the like are removed from the flakes, together with the removal of label residues, in a multi-stage fraction washer. Passage through a washing vessel and a suspension vessel is followed by a further separation stage by means of a hydrocyclone arrangement in order to separate what is by far the bulk of the PET - flakes from the remaining fractions; relatively heavy metal particles which are still present are further treated and are then optionally sorted. This process is extraordinarily costly on an industrial scale, and although it can result in a high degree of separation the final quality of the flakes nevertheless leaves something to be desired.

In a process which is known from US 5 580 905 A for the recycling of plastics waste which contains PET materials amongst others, a slurry is produced using a caustic soda solution without any mechanical washing treatment, and is heated in an environment of low oxygen content to the distillation temperature of the polyols, in order to obtain polyester salts.

In a process which is known from DE 195 45 357 A for the re-utilisation of comminuted plastics materials, water-washing and sink-float separation or hydrocyclone separation are employed. During washing and/or separation, a hydrocarbon-based organic washing medium which has a defined density is used, for example a mineral oil fraction and/or a raffinate fraction therefrom, optionally with polar solvents.

It is known in principle that PET bottles can be cleaned at an elevated temperature for 20 minutes, using a cleaning solution which contains caustic soda, wherein, however, the sensitivity of PET bottles (stress cracking and shrinkage phenomena) means that restricted upper limits have to be adhered to as regards the concentration of the cleaning solution, the treatment temperature and as regards the treatment time in particular. These constraints result in a degree of purity which would be too low for the recycling of PET flakes for the production of new bottles.

The underlying object of the present invention is to provide a process of the type cited at the outset, and an apparatus for carrying out the process, with which flakes of high quality, which can even be used for the manufacture of new bottles from said flakes, can be obtained without costly subsequent processing steps.

The aforementioned object is achieved by a process having the features of claim 1 and by an apparatus having the features of claim 13.

Because the flakes are treated for more than 20 minutes, at least in the washer, with a cleaning solution which contains caustic soda at an elevated temperature above 70°C, and are thus also processed mechanically and hydraulically at the same time, even the label adhesives which are currently on the market can be removed from the flakes, so that only a low level of subsequent treatment is required, if at all, in order to be able to process the flakes directly. The surface quality of the flakes is outstanding. The cost of washing the flakes is in fact relatively high, both as regards the process and as regards the apparatus, but this is more than offset by the high final quality and surface quality of the flakes, and the omission of costly, additional processing stages constitutes a definite advantage over conventional treatment processes, because particular emphasis is placed on the initial phase of the treatment process.

The apparatus is designed from the outset for an extremely long treatment time, for high treatment temperatures, and for the use of a cleaning solution which contains caustic soda, so as to be able to subject the flakes to mechanical, hydraulic, and intensive thermal treatment.

Particularly satisfactory results are obtained by employing a treatment time of about 30 minutes at 80 to 90°C. Higher temperatures can also be used, however, particularly if reduced pressure is employed.

In order to utilise process technology parameters and knowledge from the cleaning of PET bottles, a treatment time is employed which is prolonged by up to 50 per cent compared with the treatment time which is employed for the cleaning of PET bottles, an elevated treatment temperature is employed compared with the treatment temperature which is possible for the cleaning of PET bottles, and a higher concentration of cleaning solution is also optionally employed.

The surface of the flakes is cleaned very intensively due to the mechanical stirring and to the simultaneous subjection of the flakes to hydraulic high-pressure spraying.

It is advantageous if sieving and/or filtration is effected in the washer, or at least in a subsequent processing stage which is coupled to the washer in the process. Parts of labels, fibres and pieces of foil, as well as particles of dirt, can thereby be separated from the flakes and can be removed from the washing liquor.

At least one additive can be added to the cleaning solution in order to intensify the cleaning effect.

Due to the further treatment of the flakes in a float/sink separator with the cleaning solution which contains caustic soda, the surface quality of the flakes which is achieved using a long treatment time is so good that the flakes require no appreciable further processing.

Due to the subsequent intensive washing in an intensive washer, likewise with the use of the cleaning solution which contains caustic soda, even tenacious iridescent stains and coatings are reliably removed.

The treatment in the float/sink separator or in the intensive washer is advantageously carried out with recirculation or partial recirculation, in order to achieve a sufficiently long treatment time and an increased throughput with a more intensive cleaning effect.

Following the intensive washing stage, the cleaning solution which contains caustic soda is separated and recycled, so that the cleaning solution can be reused repeatedly with relatively small losses.

In this respect, it is advantageous if the concentration of the cleaning solution is continuously monitored, and is adjusted by metered additions of caustic soda solution at least.

The elevated treatment temperature is advantageously achieved by heating the washer.

The washer advantageously contains sieve plates and automatic filters in order to detach and reject label constituents, fibres, pieces of foil and particles of dirt.

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In order to maintain a treatment temperature between about 80 and 90°C, even under conditions of high throughput or when the washer at least is of large capacity, it is advantageous if at least one heat exchanger and/or a direct heater device is provided in the heater device, wherein the temperature can be adjusted and maintained in a particularly sensitive manner by employing an electro-pneumatic control system.

The use of a high-pressure pump and nozzle pipes results in the high-pressure cleaning of the entire surface of the flakes. The sieve plates, filters and nozzle pipes are advantageously fixedly disposed in relation to a plurality of stirrer stages, so that the mixture in the washer is also uniformly and intensively agitated.

Due to the coupling of the sink/float separator to the washer in the process, the hot cleaning solution which contains caustic soda is also used in the separator.

In a downstream intensive washer through which the flakes and a partial stream of the cleaning solution are conveyed, e.g. by a pump, an additional surface cleaning effect is ensured in which even tenacious iridescent stains and coatings are reliably removed. A recycle section can be provided between the intensive washer and the float/sink separator, in order to create at least partial recirculation and to achieve the requisite hold-up time at high throughputs without employing vessels of extremely large volume.

Following the intensive washer, the cleaning solution which contains caustic soda is separated in a separator and is recycled to the preceding process stages. For this purpose, a supply branch can be provided which leads to the washer and which advantageously comprises at least one supply pump.

At least one caustic soda measuring and metering unit should be present in the feeder device. This measuring and metering unit is connected to the supply branch or is connected thereto in some situations, but can optionally also be connected to the float/sink separator so as to be able to adjust the concentration there or even to make individual adjustments there.

So as not to adversely affect the treatment temperature, particularly in the washer, during the feeding of caustic soda and/or additives, a preheater device should be provided in the metering unit.

After the cleaning solution has been separated, it is advantageously passed through a neutraliser which is supplied with fresh water and with an acid, e.g. phosphoric acid or  $\text{CO}_2$ , with corresponding metering, and the PET material is neutralised. Rinsing with fresh water can even be carried out here for cleaning purposes.

The float/sink separator is advantageously also supplied with fresh water, in order deliberately to set a concentration which is different from that in the washer, or specifically to compensate for losses.

In order to separate at least part of the heavy material which may be contained, a heavy material separator should be disposed upstream of the washer.

The washer is advantageously designed with an uptake and throughput capacity for a treatment time of more than 20 minutes, preferably about 30 minutes.

The process according to the invention, in combination with a treatment apparatus, is explained below with reference to the drawings, where:

Figures 1A + B viewed jointly, represent a block flow diagram of an apparatus for the treatment of PET flakes; and

Figure 2 shows an enlarged portion of the apparatus of Figure 1.

A recycling apparatus or installation as shown in Figure 1A and Figure 1B, for carrying out a treatment process for conditioning at least PET constituents of plastics beverage bottles, comprises a multiplicity of stations which are explained below in relation to the successive process steps.

The beverage bottles are conveyed in stream A via a conveyor belt 1 with an associated metal separator 2 into a cutter mill Z (a dry or wet mill) and are ground or comminuted. The mixture of particles is fed via a conveying device 3 (a suction and conveying device) to an air classifier unit B, from which, via a branch line 4 and an auxiliary device 5, classified fractions, particularly light components such as a label constituents, foil constituents and the like (up to about 80 per cent thereof) and loose particles of dirt, dust and the like, are separated from PET constituents. From the air classifier unit B, the PET constituents (flakes) are fed to a heavy material trap C which is associated with a washer W. Pieces of metal, glass, stone and the like are separated in the heavy material trap C. The flakes are mixed in the heavy material trap C with a cleaning solution from a supply branch 21, wherein the cleaning solution contains caustic soda and optionally contains suitable additives. In the heavy material trap C, the cleaning solution is already at an elevated temperature, e.g. between 80 and 90°C. Thereafter, the mixture flows through the washer W for more than 20 minutes, preferably about 30 minutes. Downstream of the washer W, a float/sink separator T is provided, the feed to which can be adjusted via a shut-off element. In the float/sink separator T, floating components such as bottle cap constituents, labels, fibres, foil constituents and the like are removed from the PET flakes, as are PP and PVC components which are collected at 9 via a branch line 8. From the float/sink separator, the PET flakes, together with the cleaning solution, are fed via a branch 10 into at least one intensive washer D, from which they are fed via a branch 12 into a liquid separator E. A bypass 11 is provided between the branch 12 and a lower inlet of the float/sink separator T, in order to create a recirculation or partial recirculation between the float/sink separator T and the intensive washer D, if desired. In the liquid separator E, the cleaning solution is separated from the flakes and is recycled via the supply branch 21 to the washer W or the heavy material trap C. From the liquid separator E, the PET flakes are fed to a neutraliser G.

A caustic soda measuring and metering unit F is associated with the washer W and with the float/sink separator T. This measuring and metering unit may comprise a caustic soda container 14 with a heater device 15 in a collecting vessel 13, for example, wherein the collecting vessel 13 is connected to the supply branch 21 via a connection line 19 and a mixing device or overflow 20. Unit F is completed by a plurality of shut-off elements, and optionally by a filter and a supply pump in a metering sub-assembly 16, which is connected directly to the float/sink separator T via a line 17. A fresh water line 18, and a branch line 22



which comprises a shut-off element, lead from the supply branch 21 to the float/sink separator T (the description of Figure 2 provides further explanations of the detailed construction of the washer W and of the float/sink separating device).

The neutraliser G (Figure 1B), which is constructed in the manner of an ascending conveyor with an internal stirrer and a feed hopper fitted with a stirrer and a waste water overflow, is connected to the fresh water feed 18 and at the same time is connected via a line 23 to a measuring and metering station H, in which pH measurements can be made and additions of a neutralising acid (e.g. phosphoric acid or  $\text{CO}_2$ ) can be adjusted. The neutraliser G is followed by a mechanical dryer J, downstream of which at least one further air classifier unit K is disposed, from which a classified fraction is separated via auxiliary devices 5. Pieces of metal and small pieces of PET are separated from the stream of flakes in a downstream metal separator in a branch 24. Following this, a plurality of sorters L is provided for sorting and rejecting coloured PET constituents and PP constituents, for example, before subsequent thermal drying of the clear PET flakes is effected, by an indirect gas burner for example, in an after-dryer M. Following this, the conditioned PET flakes are either temporarily stored in a silo N or are packed in a packing station O. If waste water is formed during the process, or in order to stop the apparatus, waste water lines Y are provided for this purpose.

As shown in Figure 2, the washer W comprises a vessel 25 in which a plurality of stirrer stages 26, which have a horizontal stirrer shaft, for example, and which are fitted with blades, are accommodated spaced apart from each other and are driven by a stirrer motor 27. Fixed nozzle pipes 28 are mounted between the stirrer stages 26, and are supplied with the cleaning solution from a high-pressure pump 29 via a filter 30 and a heat exchanger WT of a heater device EP. Sieve plates (not illustrated) and automatic filter devices (not illustrated) can be provided in the vessel 25, in order to purify the cleaning solution as it is fed to the high-pressure pump 29. A defined treatment temperature is set and maintained in the washer by means of the heat exchanger WT, wherein the temperature ranges between about 80 and 90°C. A uniform treatment temperature can be obtained, for example, by employing an electro-pneumatic temperature control system. A direct heater (not illustrated) could be provided in the vessel 25 instead of the heat exchanger WT.

In the washer W, the flakes and any pieces of plastics which are still present, are intensively treated mechanically and hydraulically by the stirrer stages 26 and by a high pressure spray from the nozzle pipes 28. The treatment time is about 30 minutes. In the course of this procedure, the entire surface of the flakes is intensively cleaned.

In the subsequent float/sink separator T, floating pieces are removed. Since the float/sink separator T is connected in the process to the washer W, a hot cleaning solution which contains caustic soda is also employed therein. At the long treatment time employed, which is likewise about 30 minutes, an extraordinarily good surface quality of the flakes is achieved, so that only a slight extent of subsequent processing is necessary, if at all. An intensive separation of adhesives also occurs in the float/sink separator, wherein separation in the cleaning solution is facilitated by the different specific gravities of water and caustic soda solution.

The float/sink separator T, which comprises a vessel 31 of large volume, may contain conveyor belts and separator belts 32, 33, separating sections 34, and an inclined conveyor belt 35. A level controller 36 for the level in the float/sink separator T should also be provided. In the downstream intensive washer D, which is constructed with a housing 37 resembling an upright column with an ascending meander-shaped conveyor part comprising separating sections 38, additional surface cleaning of the PET flakes is ensured in order reliably to remove tenacious iridescent stains and coatings, for example. A partial stream at least is optionally recycled via the bypass 11, in order to make repeated use of the separator T and of the intensive washer D, for example. The downstream liquid separator E may contain sieve plates 39, for example, which allow the PET flakes to pass to the inlet of the neutraliser G but which separate the cleaning solution, which is recycled to the heavy material trap C by a supply pump 40 and supply branch 21.

The measuring and metering unit H of the neutraliser G comprises appropriate measuring devices, shut-off elements and filter groups 41, in order to feed in phosphoric acid and/or CO<sub>2</sub> depending on the measured pH. The PET flakes are neutralised in the neutraliser G with the addition of acid or of CO<sub>2</sub>, and are finally rinsed clean with fresh water in a plurality of stages.

A treatment time of 20 minutes, using a cleaning solution containing caustic soda, has proved to be necessary for the cleaning of PET bottles, in order to remove the bulk of the flavouring substances and contaminant substances which are forced into the surface. During the cleaning of PET bottles, however, the treatment time and a treatment temperature are subject to an upper limit, because stress cracking and the shrinkage of the material must be avoided. This also restricts the permissible concentration of the cleaning solution, so that during the cleaning of PET bottles the label adhesives which are currently on the market cannot be removed from the PET material. Basic knowledge from the cleaning of PET bottles is, however, used in the process according to the invention for the recycling of PET flakes, wherein defined process parameters are increased considerably as regards a defined final quality of the PET flakes compared with the purity which can be achieved for the cleaning of PET bottles.